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TECHNICAL NOTE 2788

EFFECTS OF SOLVENTS IN IMPROVING BOUNDARY LUBRICATION

OF STEEL BY SILICONES

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NACA

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SUMMARY

Because, of the known synthetic fluids, silicones best satisfy the viscometric requirements for lubricants for turbine engines, a study was conducted to establish the effect of solvents on boundary lubrication by silicones. Boundary-lubrication data were obtained which are considered substantiating evidence for a hypothesis that, in solutions of solvents blended with silicones, the silicones form a closely packed and oriented adsorbed film on ferrous surfaces. The solutions reduced friction and prevented surface failure even when the solvent as well as the silicone was an extremely poor lubricant. These data indicate that satisfactory lubrication is the result of a solvation effect rather than a lubrication additive effect of the solvent because 30 to 50 percent of solvent was necessary for good results. The best results were obtained with solvents having dipole moments.

Solutions of di(2-ethylhexyl) sebacate and di(2-ethylhexyl) adipate in silicone fluid were found to have viscometric characteristics approaching that of the silicone alone and were effective lubricants at temperatures above 300° F. The temperature limit of effective lubrication could be increased by employing silicones of greater average chain length.

INTRODUCTION

New turbine engines for military aircraft have lubrication requirements that cannot be met satisfactorily by available petroleum lubricants. In particular, extremely good viscometric and thermal stability properties are necessary for lubricating fluids; the specific requirements are set forth in a recent military specification, MIL-L-7808. Viscosity at -65° F must be sufficiently low to allow adequate pumpability of the fluid. Also, the fluid should be thermally stable so that harmful decomposition and vaporization will not occur. Petroleum lubricants are not satisfactory with regard to either low-temperature viscosity or high-temperature thermal stability. A number of tailor-made synthetic fluids are available which satisfy the viscometric and thermal stability requirements and, consequently, have promise as lubricants for aircraft turbines.

Silicone fluids are one class of synthetic lubricants that are very interesting, primarily, because they have the best viscometric properties of any known compounds. These fluids have two basic limitations: inadequate oxidation resistance or thermal stability and poor lubricating effectiveness. A private communication from a manufacturer of silicones indicates that the problem of increasing oxidation resistance and thermal stability is much less involved than that of improving lubrication. The manufacturer stated that it is possible to supply silicones with greatly increased oxidation resistance at 500° F without adversely affecting viscometric properties. The principal characteristic that has limited consideration of silicones as lubricants for turbine engines, however, has been that they are notoriously poor boundary lubricants for ferrous surfaces. A classified research report by E. D. Tingle of the Royal Aircraft Establishment discusses the mechanisms of lubrication by silicones. The research reported herein, which was conducted at the NACA Lewis laboratory, is concerned with the behavior of silicone lubricants in boundary lubrication of ferrous surfaces.

Two hypotheses have been considered as possible explanations for the poor lubricating abilities of silicones. Substantiation of either or both hypotheses will provide basic information necessary for selection of materials for blending with silicones to make them more effective lubricants.

Boundary-lubrication experiments were made using a kinetic friction apparatus. Ten different solvents of varied chemical structure were studied in various concentrations in the silicone. Two of the solvents were studied at high temperatures. These were selected on the basis of their probable effect on the silicone lubrication hypotheses described in the next section.

WORKING HYPOTHESES

It appears logical to preface a discussion of the working hypotheses with a consideration of the silicone molecule. This molecule, as shown in table I, is composed of a skeleton linear structure of alternate silicon and oxygen atoms with each silicon atom also bonding two methyl groups in order to satisfy its chemical valence of four. Each end of the chain is terminated by a silicon atom with three methyl groups attached. The bond between the silicon and oxygen atoms has a strongly ionic character, while the silicon to carbon bonds are predominantly covalent. The silicon-oxygen-silicon bond angle is surprisingly large and has been estimated as being $160^\circ \pm 15^\circ$ (reference 1) leading to a very mild zigzag geometry in the chain itself. The dipole moment of the silicone molecule has been calculated to be approximately $0.7\sqrt{Q}$ (reference 1) where Q represents the number of silicon-oxygen groups in the chain.

Studies of the force-area curves of a monomolecular silicone oil film spread on a water surface (reference 2) show that at low film pressures the chain lies flat on the surface with each silicon and oxygen atom oriented in the water-oil interface. All the silicon atoms will then lie in the same plane with all the methyl groups on one side of that plane. As the film pressure is increased, the orientation changes so that every other silicon or oxygen atom lies in the interface. Further compression causes the chain to coil into helical form with each turn of the helix having six monomer (silicon-oxygen) units. The energy of adhesion is naturally at a maximum when the chain is uncoiled and adsorbed with its long axis parallel to the water surface. Reference 3 indicates that the addition of a solvent will also cause the helical chain to uncoil.

Therefore, it appears that, in bulk solution, similar structural effects occur. Here, the silicone molecule assumes a helical configuration and the adhesional energy of the molecule to a metal surface is at a minimum. Addition of a solvent, however, analogous to the act of decreasing the film pressure in the force-area studies, allows the molecule to uncoil until it reaches a point where the energy of adhesion of the molecule to a solid surface reaches a maximum value, thus it may be hypothesized that the addition of a solvent provides an oriented layer of silicone molecules which may act as an effective film for boundary lubrication.
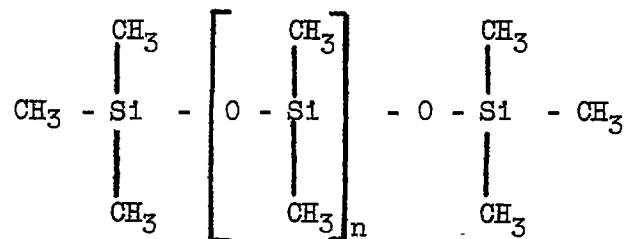
The primary hypothesis considered herein is that the use of solvents will influence lubricating ability because dilution and solvation will affect the molecular arrangement of silicone fluids. Several investigators have shown straight-chain polymers to assume characteristic shapes that are dependent on the type of medium in which they are dissolved. Since molecular arrangement and orientation effects are known to be significant factors in boundary lubrication, the influence of the type of solvent in improving the lubricating effectiveness of silicones may be of great importance.

A secondary hypothesis, applicable to lubrication by silicones, is concerned with the availability of oxygen for the maintenance of a protective oxide layer on steel surfaces. Reference 4 shows that oxide films on steel exert an important influence on frictional behavior of lubricated and unlubricated surfaces. The second hypothesis suggests that silicones prevent available oxygen from reacting with ferrous surfaces to form oxide films. This could occur either by preferential oxidation of the silicone or by the lack of free oxygen in the fluid and the possibly impervious (to oxygen) nature of the boundary film formed by silicones.

EXPERIMENTAL FLUIDS

Silicones

The silicone oils used for all tests in this investigation were polydimethyl siloxane polymers of the following structure:

$$CH_3 - \underset{\underset{CH_3}{|}}{\overset{\overset{CH_3}{|}}{Si}} - \left[O - \underset{\underset{CH_3}{|}}{\overset{\overset{CH_3}{|}}{Si}} \right]_n - O - \underset{\underset{CH_3}{|}}{\overset{\overset{CH_3}{|}}{Si}} - CH_3$$

These fluids were obtained from the Dow Corning Corp. (DC 200 series) in two different viscosity grades, 10 centistokes and 50 centistokes at 25° C, and were utilized without any further treatment.

Solvents

The various solvents used in the research reported herein, their characteristic chemical structures, and the procedure followed in their preparation for friction experiments are shown in table I. When known, their viscosities are also reported.

The freedom of nonpolar solvents from impurities was checked by running them as lubricants. Severe failure at low loads in friction experiments was taken as an indication of the absence of polar impurities, a procedure which has been found very successful in demonstrating the purity of nonpolar compounds.

APPARATUS AND PROCEDURE

A diagrammatic sketch of the apparatus used to study the boundary lubrication effectiveness of the solutions of silicone with various solvents is shown in figure 1. The basic elements of this apparatus were the rotating, mild-steel disk specimen ($2\frac{1}{2}$ in. diam) and the cylindrical hardened (Rockwell C-60) SAE 1095 steel rider specimen with a hemispherical ($\frac{3}{16}$ in. rad) contact tip. The rotating specimen was driven through a belt system by an electric motor coupled with a variable-speed power transmission device. This arrangement allowed good speed control with a range of sliding velocities up to approximately 500 feet per minute. The runs reported herein were made with a sliding velocity of 120 feet per minute. Loading was obtained by the use of dead weights to apply force through the pully system shown in figure 1. The net loads at the sliding contact were from 200 to 2000 grams (87,000 to 188,000 lb/sq in., initial Hertz surface stress). These loads are representative of those occurring in aircraft turbines.

The rider-holder assembly was located by rolling contact bearings with the shaft free to move axially and to rotate within the bearing

housing. The apparatus was insensitive to the very small friction losses in the bearings. Contact of the rider specimen with the disk provided axial location of the rider-holder assembly when load was applied. Friction force between the rider and the disk was measured by four strain gages mounted on a copper-beryllium dynamometer ring. The friction-force readings were obtained from an indicating-type calibrated potentiometer. The dynamometer ring restricted the angular motion of the rider-holder assembly to less than 2° as necessary to obtain strain (friction-force) measurements. The radius-of-force application by the rider specimen and the radius-of-force measurement by the strain-gage assembly were the same allowing a direct measurement of friction force. The friction coefficient μ is the ratio of friction force to applied normal load. Friction coefficients were generally reproducible within ± 0.02.

The fluid lubricants studied were contained in a pyrex cylindrical jar fastened to the bottom of the apparatus mounting plate. In most cases 300 cubic centimeters of fluid was sufficient for an experiment. In order to perform experiments at elevated temperatures (up to 500° F), the lubricant jar was surrounded by a fluid bath heated with an electric hot plate. Operation of the hot plate, which maintained temperature of the bath, was automatically controlled by a commercial capacitance-controlled millivoltmeter activated by an iron-constantan thermocouple inserted in the bath. The bath was maintained at any desired temperature within approximately $\pm 3^{\circ}$ F.

A new set of specimens was used for each experiment. The specimens were originally finished by fine turning; however, before initial use and successive re-use, the disks were refinished with abrasive cloth. This was done while rotating the specimens in a drill press by holding successive grades of abrasive against the moving surface. Coarse abrasive was used to remove the previous finish or wear track and final polishing of the disk was accomplished with grade 1/2 polishing cloth. The disk surfaces had uniform circumferential finishing marks and had a surface roughness of approximately 30 rms, as measured with a profilometer. The rider specimens were finished with grade 3/0 polishing paper while rotating in the drill press. The disk specimens had a hardness of approximately Rockwell A-50 and the rider specimens were approximately Rockwell C-60.

Prior to use, the specimens were cleaned according to the following procedure: Rust-inhibiting grease was removed from the specimens by soaking and wiping in low-aromatic cleaning naphtha. The specimens were then wiped with clean cloths saturated with either redistilled acetone or a solution containing equal parts of redistilled acetone and benzene. The specimens were then thoroughly scrubbed with moist levigated alumina powder. The alumina was removed by rinsing with tap water and the specimen was tested for cleanness by observing the ability of water to wet the surface. Water was then removed from the specimen by successive immersion and rinsing with redistilled 95 percent ethyl alcohol or redistilled acetone. In most cases the specimens were used immediately after cleaning;

in some cases, however, they were stored in a laboratory desiccator for several hours before use.

For all runs with each solvent, the procedure to show the effect of solvent concentration included successive series of friction runs with increased solvent concentration using the same sample of silicone fluid. The total volume of lubricant (solution) was the same in all experiments. In order to obtain each individual datum point, the apparatus was started with the surfaces in contact at the desired load; as soon as the apparatus was at the proper speed (120 ft/min) and the friction readings were stable, data were taken and the drive motor shut down. Unless some interesting or unusual data were observed where prolonged operation was advantageous, the total operating time to obtain each friction measurement was less than 1 minute. With each solution, runs were made at progressively greater loads. Elevated temperature runs consisted of a similar series of runs made at varied temperature levels as high as 500° F. Subsequent series of runs were made at higher temperature levels until lubrication failure was experienced, at which point the experiment was generally terminated. Lubrication failure was easily identified by increased friction coefficient, audible chatter of sliding specimens, and visible surface welding.

RESULTS

Room-Temperature Lubrication Experiments

Friction data for the 50-centistoke silicone fluid is presented in figure 2. As evidenced by high friction and surface welding, the silicone did not provide effective lubrication under any of the conditions of this experiment. Effective lubrication is defined as a condition where there is no surface welding and where friction coefficient is in a range common to boundary lubrication. Varying the silicones (10 cs fluid and 50 cs fluid) had no discernible effect on the friction, wear, and surface-failure properties. Progressive failure of the wear track caused friction to be slightly higher than reported in reference 5. Besides the high friction, very rapid wear of the sliding surfaces was noted. Accumulation in this fluid of very fine wear debris caused the fluid to be practically opaque to light before the completion of the experiment. The stability of the suspension (of wear debris) in the fluid indicated that wear-debris particle size at least approached the colloidal range. After the experiment, the slider surfaces showed the expected evidences of extreme surface failure with appreciable surface welding and very rapid wear.

In order to establish whether or not the second hypothesis was correct, wear products obtained during lubrication experiments with pure silicone were studied by X-ray diffraction techniques. The wear debris was found to be primarily α-iron. With more effective lubricants such as petroleum, wear debris can usually be identified as a mixture of oxides of iron. These results are of a preliminary nature and further study is required to provide adequate verification for the hypothesis on the role of oxygen in lubrication by silicones.

Friction data obtained by adding varied amounts of the solvents previously mentioned to the basic silicone fluid are presented in figure 3. With benzene alone, surface welding occurred at all loads. The data obtained by the addition of benzene to the silicone oil are shown in figure 3(a). A concentration of 9 percent by volume of benzene raised the friction value of the silicone. The next higher concentration was only slightly effective in lowering the coefficient of friction. At a concentration of 29 percent, a strong effect was noted at the lower loads with film failure occurring when the load was increased. At approximately 38 percent by volume of solvent concentration, an optimum point was reached. The optimum was defined as the minimum concentration that would give effective lubrication. Solvent concentration was held as low as possible in selecting the optimum so that the desirable viscosity characteristics of silicones could be retained. The effect of a very large concentration of solvent (benzene) in silicone on friction behavior is shown in figure 3(b).

In order to determine the effect of structural similarity, the structural analog of benzene, cyclohexane, was used to obtain the data shown in figure 3(c). It was noteworthy that the cyclohexane had only a slight effect on friction which was more apparent at the lower loads; any film which might have formed apparently ruptured at higher pressure. In fact, for a solvent concentration of 50 percent the results are poorer than for silicone alone.

In figure 3(d) are shown the results for cyclohexene, another compound similar to benzene, except that it has a permanent dipole moment. These results may not be a clear illustration of solvent effect since the cyclohexene is a reasonably good lubricant in its own right. Tetralin (fig. 3(e)) followed the same general trend as cyclohexene and alone also showed good lubricating properties. Toluene (fig. 3(f)), the next solvent, behaved similarly to benzene. The structural analog of toluene methyl cyclohexane, (fig. 3(g)) failed to show any marked improvement; this failure to show marked improvement was anticipated from the results of the benzene and cyclohexane additions. Methylethyl ketone (fig. 3(h)) produced results similar to benzene except that the optimum concentration was slightly lower.

In figure 3(i) are shown the effects of adding another solvent, carbon tetrachloride. It is noteworthy that much smaller concentrations were needed to provide adequate lubrication, but this apparent exception can be readily explained on the basis of the well-known "E.P." (extreme pressure) effect for carbon tetrachloride which probably masked any solvent-dilution effect and in itself became the dominating factor.

Because lubricant blends of silicone and volatile solvents could hardly be considered as satisfactory engine lubricants, a series of runs was made using two diesters, di(2-ethylhexyl) sebacate and di(2-ethylhexyl)

adipate (figs. 3(j) and 3(k)). These diesters have already been widely accepted as satisfactory lubricants for current aircraft turbine-type engines and in these experiments were used as the "solvents" for silicones. It was felt that these combinations would provide practical lubricants of low volatility that might have viscometric properties approaching those of the pure silicone (fig. 4) and with effective lubrication resulting from a possible "solvent" effect of the diesters.

It was found that the 50-centistoke silicone would not dissolve more than 15 percent by volume of the sebacate diester. An equal mixture of 50-centistoke silicone with 10-centistoke silicone (producing a fluid roughly approximating the commerical 20-cs silicone) was capable of easily dissolving 33 percent of di(2-ethylhexyl) sebacate. Although the adipate diester was approximately three times as soluble as the sebacate in the silicones, some solutions were made from the same mixture of silicones as was used with the sebacate. Any lubrication advantage obtained by increasing the diester concentration would be offset by less desirable viscometric properties.

Friction data obtained with 10- and 33-percent solutions of sebacate diester in silicone fluids are presented in figure 3(j). The 10-percent solution was made with 50-centistoke silicone and the 33-percent solution was made using equal parts of 10- and 50-centistoke fluids. The friction curve for the pure 50-centistoke fluid given in figure 2 also satisfied the data obtained with 10-centistoke fluid and the mixture of 10- and 50-centistoke fluid. Figure 3(j) shows that the higher concentration of diester was necessary to obtain low friction and to prevent surface failure. The friction values obtained for the 33-percent solvent solution were only slightly higher than those for the pure di(2-ethylhexyl) sebacate. A check with the pure adipate and with a solution of 33-percent adipate in 10-centistoke silicone fluids produced results (fig. 3(k)) similar to those obtained with the sebacate diester.

Elevated-Temperature Lubrication Experiments

As previously stated, one of the most important reasons for using the diesters as "solvents" was to obtain continued effective lubrication at high temperatures. Accordingly, a series of friction experiments was made at various temperature levels with several diester-silicone solutions (fig. 5). These experiments were made at temperatures higher than the present operating levels for bearings of aircraft gas turbines.

The limiting temperatures used in some cases are a function of the apparatus (fluid bath) and where failure was not observed do not necessarily imply a maximum operating temperature characteristic of the lubricant studied. The results showed that di(2-ethylhexyl) sebacate alone (fig. 5(a)) was effective at the most severe operating conditions

used. The mixture of di(2-ethylhexyl) sebacate with silicones (fig. 5(b))
was also an effective lubricant at the most severe conditions of the
experiment with no evidence of surface failure and with friction values
which were approximately the same as those obtained at room temperature
(fig. 3(j)).

Although not experimentally established, it is believed that the
di(2-ethylhexyl) sebacate alone would continue to be an effective lubri-
cant at considerably higher temperatures than those reported herein.
Also, it is probable that the solution of silicone and diester was run
at very nearly its limiting temperature since friction values were becom-
ing more unstable.

Friction data for di(2-ethylhexyl) adipate at the maximum elevated
temperatures (fig. 5(c)) are almost identical with those obtained at room
temperature (fig. 3(k)). Figure 5(d) shows unstable friction values for
the solution of one-third adipate in equal parts of 50- and 10-centistoke
silicones at a temperature of 302° F with a load of 1600 grams. When the
less viscous silicone was used with the adipate solvent, unstable friction
values (fig. 5(e)) occurred at the same temperature but at a less severe
load (600 g) and at a lower temperature (175° F) with the same load
(1600 g). Unstable friction values in many cases may indicate incipient
lubrication failure. In no case with the diester-silicone solutions,
even where incipient surface failure may have occurred, was there a rapid
accumulation of fine particles of wear debris such as was characteristic
of silicone lubricants alone.

A summary of the results presented in the preceding paragraphs is
given in table II. The table contains data on optimum concentration, and
friction values from data curves and information on surface conditions
as observed.

Low-Temperature Physical Behavior

The sebacate-silicone fluid and the adipate-silicone fluid were
checked to establish the low-temperature stability of the solutions.
Samples of the fluids were placed in test tubes and immersed to the fluid
level in dry ice and acetone; a thermometer was inserted in the test
tubes. The fluid containing one-third di(2-ethylhexyl) sebacate and two-
thirds silicone (10 cs) became slightly turbid as temperatures were
decreased below -30° F but there was no solidification product settling
out of the fluid and this turbidity characteristic was reversible as tem-
peratures were subsequently increased to above -30° F. This fluid could
be easily poured at -100° F. The solution containing one-third di(2-
ethylhexyl) adipate and two-thirds silicone (10 cs) also showed turbidity
at -40° F and could easily be poured at -100° F. The turbidity character-
istics of the adipate-silicone fluid was perfectly reversible and apparently

had no adverse effect on the fluid. Di(2-ethylhexyl) adipate as received also had the same turbidity characteristic observed for the solution. Viscosity-temperature characteristics of a solution of one-third di(2-ethylhexyl) adipate in 10-centistoke silicone and of the individual fluids shown in figure 4 also indicate low-temperature stability of a typical blend.

Effect of Conventional Lubrication Additive

The addition of conventional lubrication additives might be a means of improving lubrication by silicones. Such additives are not generally soluble in the silicone alone but are soluble in diesters. As part of this investigation, a lubricant solution of diester in silicones was compounded which included 2 percent of the lubrication additive tricresyl phosphate. The results (fig. 6) indicate that the additive may have had a slight, if any, beneficial effect on the lubricating ability of the solution. Under the conditions of these experiments the tricresyl phosphate showed no appreciable effect; with more severe sliding conditions, the additive might improve lubrication.

DISCUSSION

The explanation of the working hypothesis concerned with the effect of solvents is apparently an over-simplification since two of the solvents used in this work had only a slight effect on the frictional properties of the silicones. It is interesting to note that these two exceptions are both saturated hydrocarbons which have neither a dipole moment nor as strong a possibility for an induced dipole.

The summary of data presented in table II indicate that the solvents are not functioning as conventional lubrication additives. All solvents had beneficial effects on friction and surface failure when blended with the silicone regardless of molecular structure or lubricating properties of the solvents alone. In all cases, except carbon tetrachloride, the optimum concentration of solvent to silicone was at least three to five times greater than is necessary with conventional lubrication additives.

Lubrication is affected by changing the silicone without changing the solvent (figs. 5(d) and 5(e)); with the use of identical volume percentages of the same solvents in silicones of different chain lengths there was a change in lubricating effectiveness. The solvent blend with silicones having a predominance of larger molecules (the higher-viscosity fluid) provided stable friction values at more extreme loads and bulk temperatures than did the solvent blend with silicones having smaller molecules (lower-viscosity fluid). If lubrication was provided by the solvent alone the change of silicones would have little or no effect on the results.

All these data, and particularly the data obtained with benzene and toluene, indicate that it is unlikely the solvents are functioning as lubricants. Benzene, which several investigators have used as an example of an antilubricant (reference 6), in the experiment reported herein provided effective lubrication when in solution with silicones. Such data indicate that a beneficial "solvation effect" may be obtained by blending two poor lubricants (solvent and silicone) and that the resulting fluid can have lubricating properties that are not dependent on the lubricating ability of either constituent.

This solvation effect may result from a combination of the following: (1) The solvent causing the silicone molecules to uncoil thus allowing them to become more closely packed, (2) uncoiling of the silicone molecule by the solvent increases the dipole moment of the silicone molecule, and (3) the addition of the dipole moment or induced dipole moment of the solvent to the dipole moment of the silicone. Polar moments give rise to molecular orientation on a lubricated surface and, with close packing, to greater lateral stability of a lubricating film.

When the solvents have a dipole moment or the possibility of an induced dipole moment, the blends with silicone provide further improvement in lubricating effectiveness over solvents which are not polar (table II). In several cases the solvents alone are effective lubricants and might possibly have masked the fundamental solvation effect described herein. If it is correct to assume that the solvents with dipole moments could act as the lubricant in the same manner as a polar lubricant additive (such as stearic acid, reference 4) the concentration required for effective lubrication would be a small fraction of that required in these experiments.

Results obtained with concentrations of solvent lower than the optimum provide further support for the solvation hypothesis. For example, with benzene (fig. 3(a)) concentrations of 29 percent and 33 percent failed to lubricate at the higher loads where the optimum solvent concentration (38 percent) was completely effective. Up to the optimum concentration, the failure load, as indicated by increasing friction values, is in direct relation to the concentration of solvent. Concentrations of benzene considerably greater than the optimum also resulted in progressively decreasing lubricating effectiveness (fig. 3(b)).

Qualitative data on lubricating effectiveness of mixtures of silicones and diesters for use in greases were previously reported in reference 7 where it is suggested that lubrication was due to the diester alone. This finding is not consistent with the results reported herein.

The data available do not make it possible to accept completely or to reject the hypothesis on the role of oxygen on lubrication by silicones.

SUMMARY OF RESULTS

The following results were observed from boundary-lubrication studies conducted with silicones containing high percentages of various solvents:

1. Boundary-lubrication data were obtained which are considered substantiating evidence for a hypothesis that, in solutions of solvents blended with silicones, the silicones form a closely packed and oriented adsorbed film on ferrous surfaces. The solutions reduced friction and prevented surface failure even when the solvent and the silicone separately were extremely poor lubricants. These data indicate that satisfactory lubrication is the result of a solvation effect rather than a lubrication additive effect of the solvent since 30 to 50 percent by volume of solvent was necessary for good results. The best results were obtained with solvents having dipole moments.

2. Solutions of di(2-ethylhexyl) adipate or di(2-ethylhexyl) sebacate in silicone fluid were prepared in an effort to obtain a practical lubricant utilizing the solvent hypothesis. The solutions were effective lubricants and were found to have viscometric characteristics approaching that of the silicones alone. The upper temperature or the load limit for effective lubrication could be increased by increasing the average chain lengths of the silicone. The solutions studied were effective lubricants at temperatures above 300° F.

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    National Advisory Committee for Aeronautics
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TABLE I - CHARACTERISTICS OF EXPERIMENTAL FLUIDS

Fluid	Chemical structure	Viscosity (a)	Special treatment
Silicone[b]	$CH_3-Si(CH_3)_2-[O-Si(CH_3)_2]_n-O-Si(CH_3)_2-CH_3$	10 and 50 cs at 25° C.	Used as received
Benzene	(benzene ring structure)	0.74 cs at 20° C	Fractionally distilled. Percolated several times through columns of silica gel and Fuller's earth.
Cyclohexane	(cyclohexane ring structure)	1.31 cs at 20° C	Same as benzene
Cyclohexene	(cyclohexene ring structure)	0.82 cs at 20° C	Same as benzene
Tetralin	(tetralin bicyclic structure)	------------------	Same as benzene
Toluene	(toluene ring structure)	0.68 cs at 20° C	Percolated several times through columns of silica gel and Fuller's earth (reagent grade).
Methyl cyclohexane	(methylcyclohexane ring structure)	------------------	Same as benzene
Methylethyl ketone	$H_3C-C(=O)-C_2H_5$	0.53 cs at 20° C	Used as received (technical grade)
Carbon tetrachloride	$Cl-C(Cl)(Cl)-Cl$	0.61 cs at 20° C	Used as received (reagent grade)
Di(2-ethylhexyl) sebacate[c]	$COOCH_2CHCH_2CH_2CH_2CH_3$ with C_2H_5 branch, $(CH_2)_8$, $COOCH_2CHCH_2CH_2CH_2CH_3$	23 cs at 20° C	Used as received
Di(2-ethylhexyl) adipate[d]	$COOCH_2CHCH_2CH_2CH_2CH_3$, $(CH_2)_4$, C_2H_5, $COOCH_2CHCH_2CH_2CH_3$ with C_2H_5	15 cs at 20° C	Used as received

[a] Handbook data.

[b] DC 200 series from Dow Corning Corp.

[c] Plexol 201 from Rohm & Haas Co.

[d] Flexol A-26 from Union Carbide and Carbon Corporation.

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TABLE II - SUMMARY OF OBSERVATIONS ON EFFECT OF SOLVENTS ON LUBRICATION BY SILICONES

Solvent	Benzene	Cyclo-hexane	Cyclo-hexene	Tetralin	Toluene	Methyl cyclo-hexane	Methyl-ethyl ketone	Carbon tetra-chloride	Di(2-ethyl-hexyl) sebacate	Di(2-ethyl-hexyl) adipate	Silicone
Optimum concentration (percent by volume)	[b]38	30	[a]50	[a]30	40	30	[a]33	[c]10	[a]33	[a]33	-----
Friction coefficient for solvent alone at 1000 g load	>0.8	>0.7	0.15	0.15	>0.8	>0.7	0.18	0.15	0.13	0.13	0.32
Friction coefficient for optimum blend at 1000 g load	0.19	0.26	0.15	0.18	0.18	0.28	0.12	0.11	0.15	0.15	-----
Surface condition after run with solvent alone	Welding	Welding	No welding	No welding	Welding	Welding	No welding	No welding	No welding	No welding	Welding (no solvent)
Surface condition after run with optimum blend	No welding	Incip-ient welding	No welding	No welding	No welding	Incip-ient welding	No welding	No welding	No welding	No welding	-----

[a]Solvent has a dipole moment.

[b]Solvent has a possibility of an induced dipole moment.

[c]Solvent has extreme pressure lubricant properties.

┌ Shaft rotation

Frictional force ─

Drive pulley

Load

Strain-gage
assembly ─

Bearing
housings ─

Cylindrical
pyrex jar ─

Weight pan

Lubricating
fluid ─

Rotating disk specimen
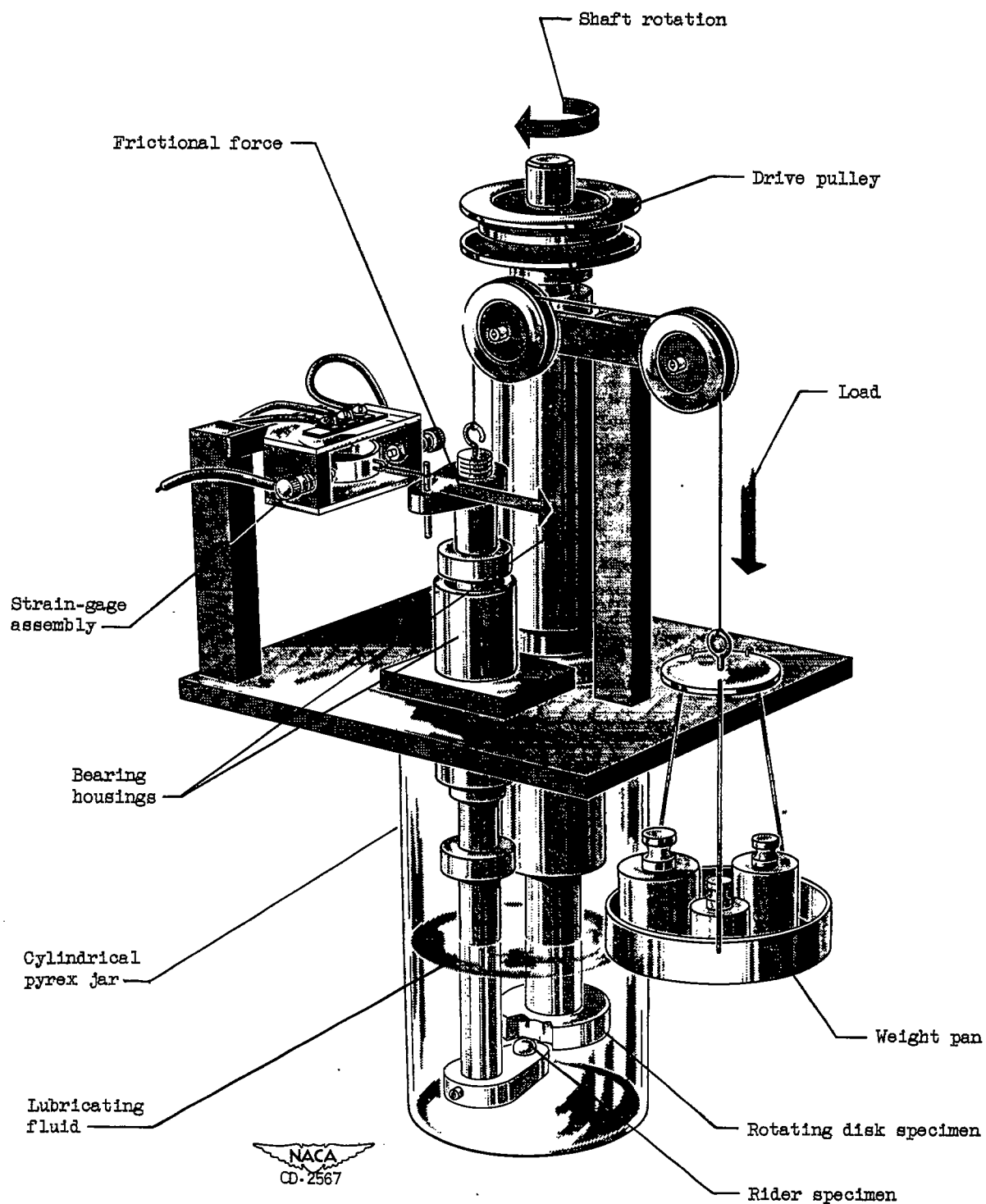
Rider specimen

NACA
CD-2567

Figure 1. - Schematic diagram of friction apparatus for studying boundary lubrication by
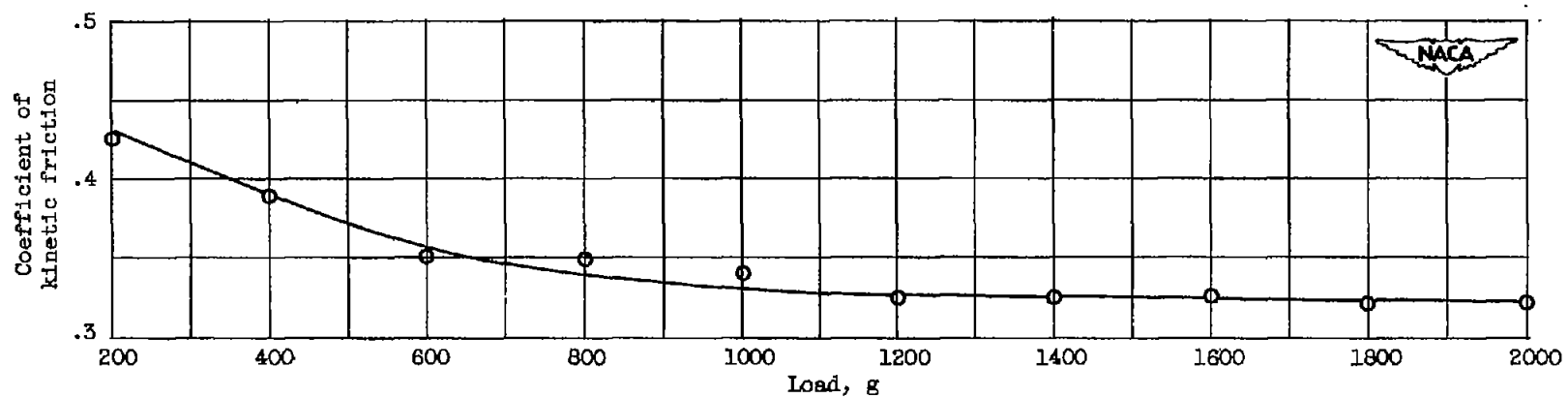bulk lubricants.

Figure 2. - Effect of load on friction of steel sliding against steel lubricated with 50-centistoke (at 25° C) silicone fluid. Sliding velocity, 120 feet per minute.
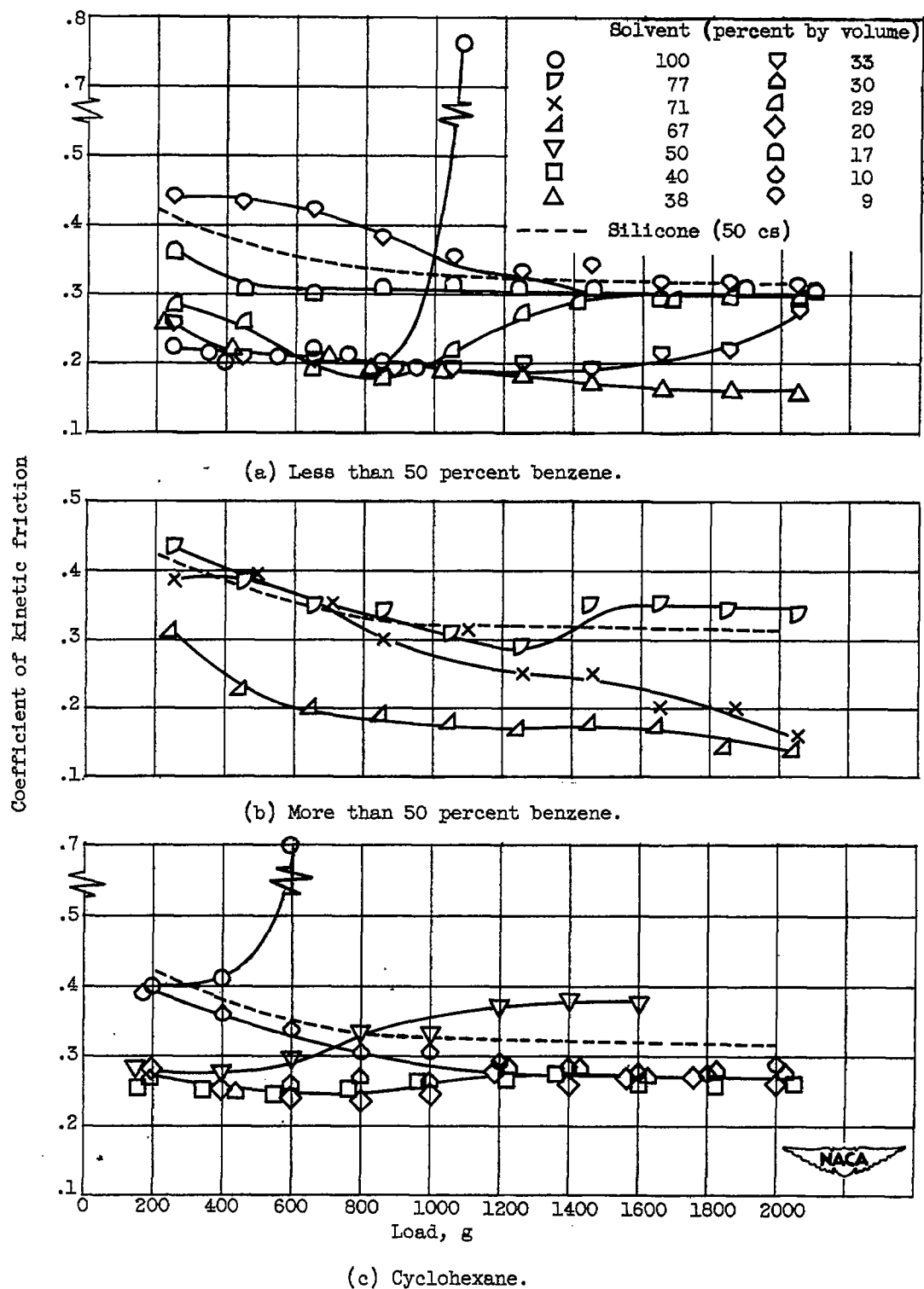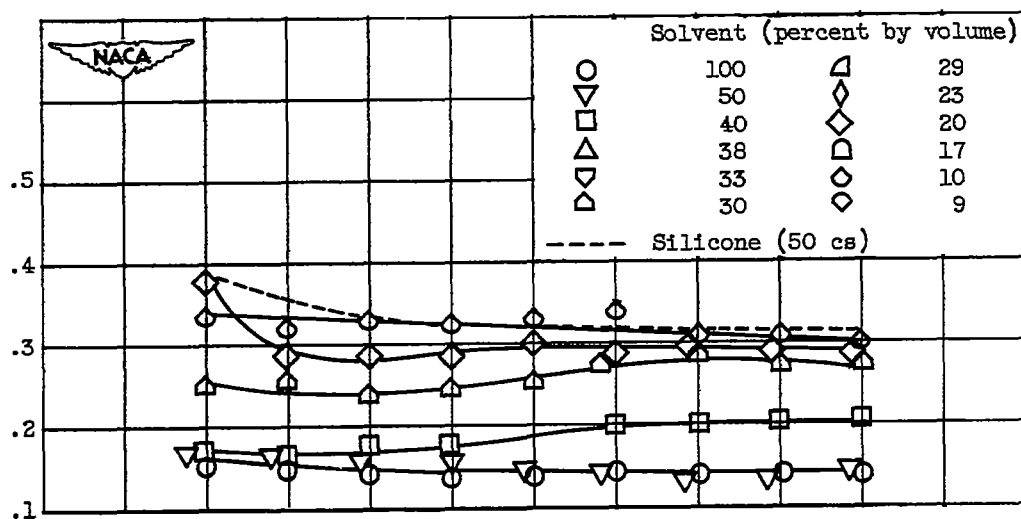
(a) Less than 50 percent benzene.
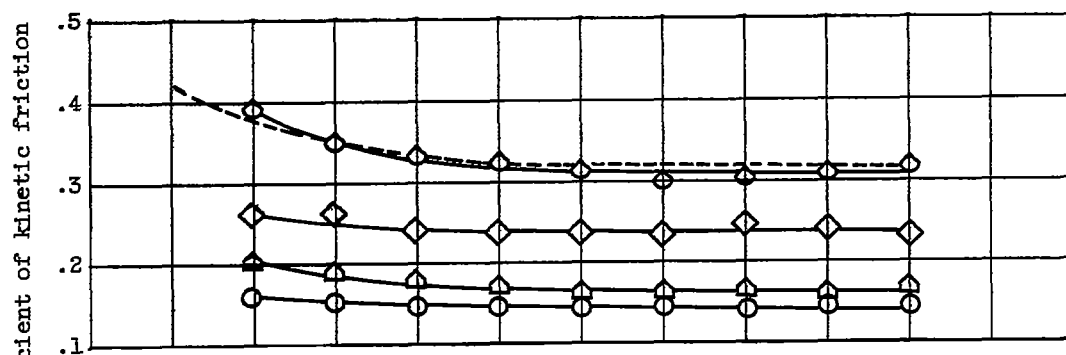
(b) More than 50 percent benzene.
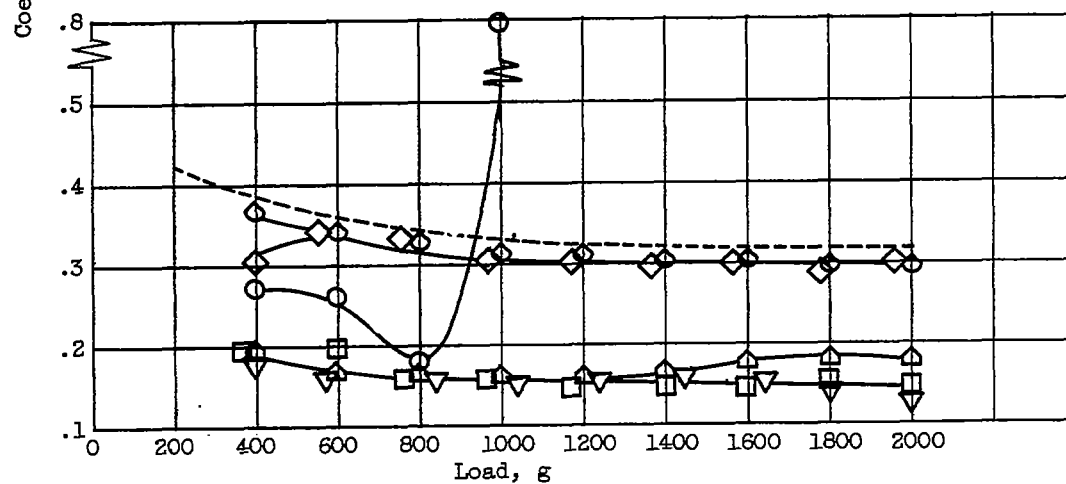
(c) Cyclohexane.

Figure 3. - Effect of concentration of various solvents in silicone (50 cs at 25° C) on coefficient of friction over a rangeoof loads. Sliding velocity, 120 feet per minute.

Figure 3. - Continued. Effect of concentration of various solvents in silicone (50 cs at 25° C) on coefficient of friction over a range of loads. Sliding velocity, 120 feet per minute.

(g) Methyl cyclohexane.

(h) Methylethyl ketone.
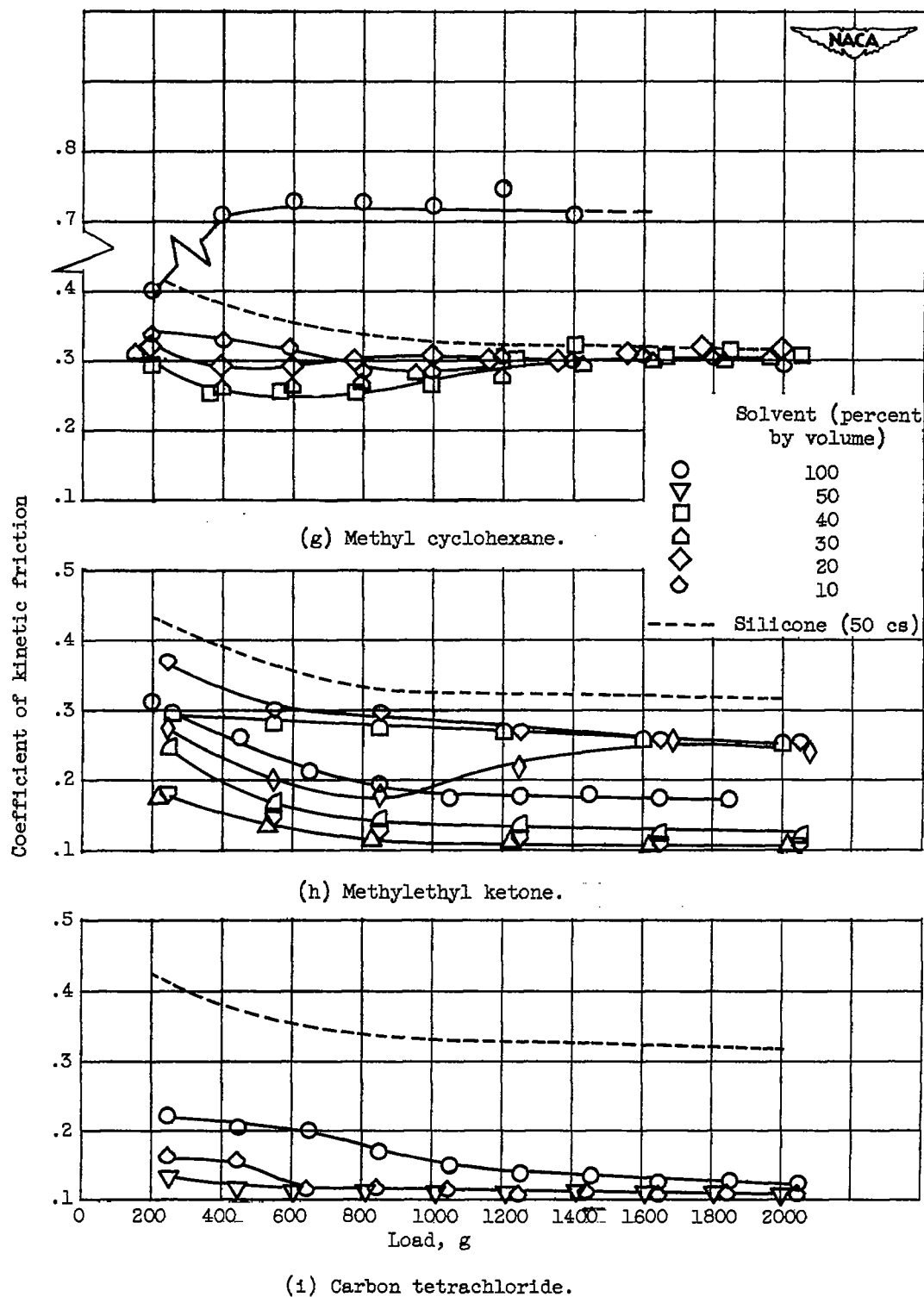
(i) Carbon tetrachloride.

Figure 3. - Continued. Effect of concentration of various solvents in silicone
(50 cs at 25° C) on coefficient of friction over a range of loads. Sliding
velocity, 120 feet per minute.

(j) Di(2-ethylhexyl) sebacate (10 percent solvent fluid prepared with
50 cs silicone and 33 percent solvent fluid was made with equal
parts of 10 cs and 50 cs silicones).

(k) Di(2-ethylhexyl) adipate (all solutions
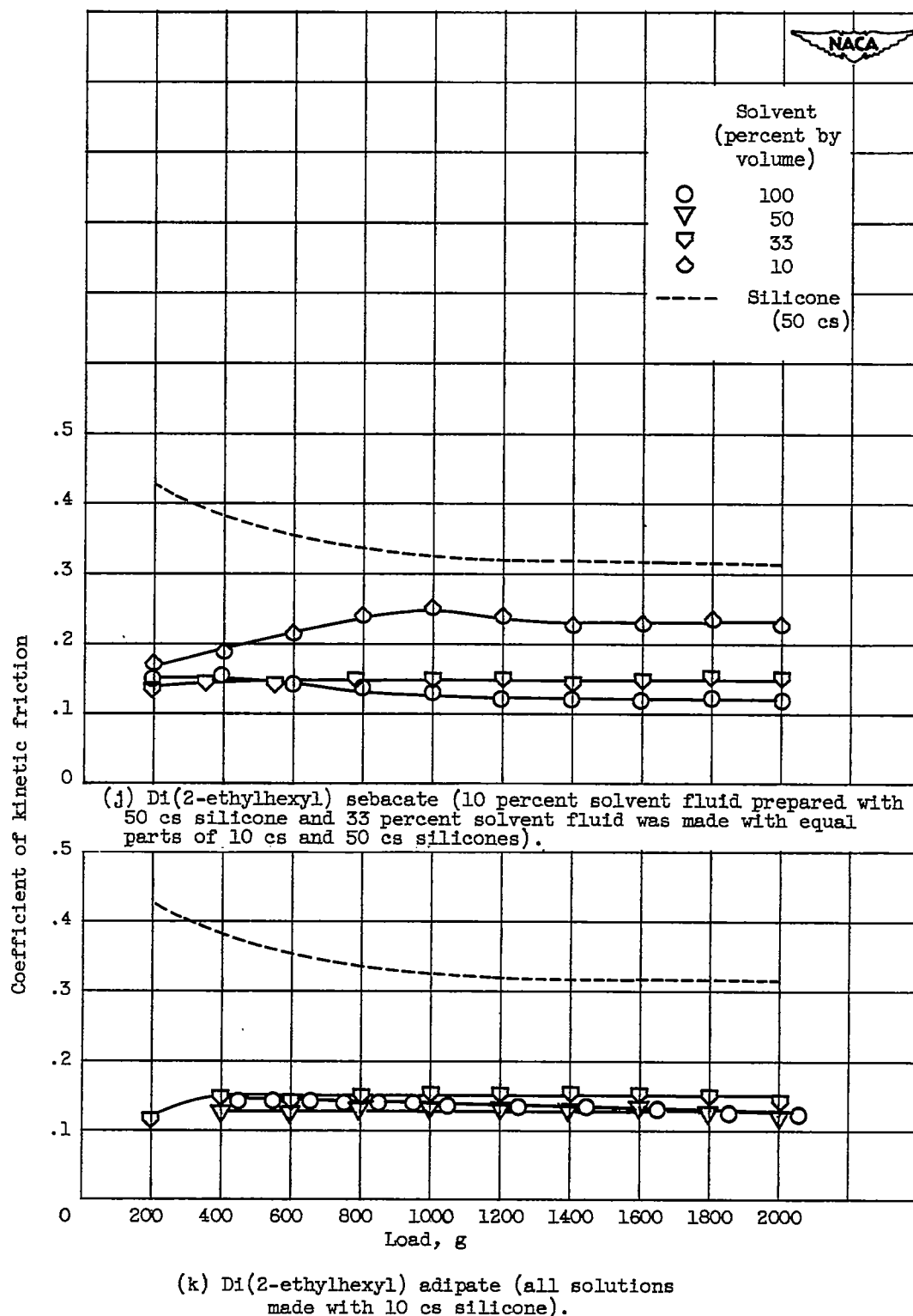made with 10 cs silicone).

Figure 3. - Concluded. Effect of concentration of various solvents in
silicone (50 cs at 25° C) on coefficient of friction over a range of
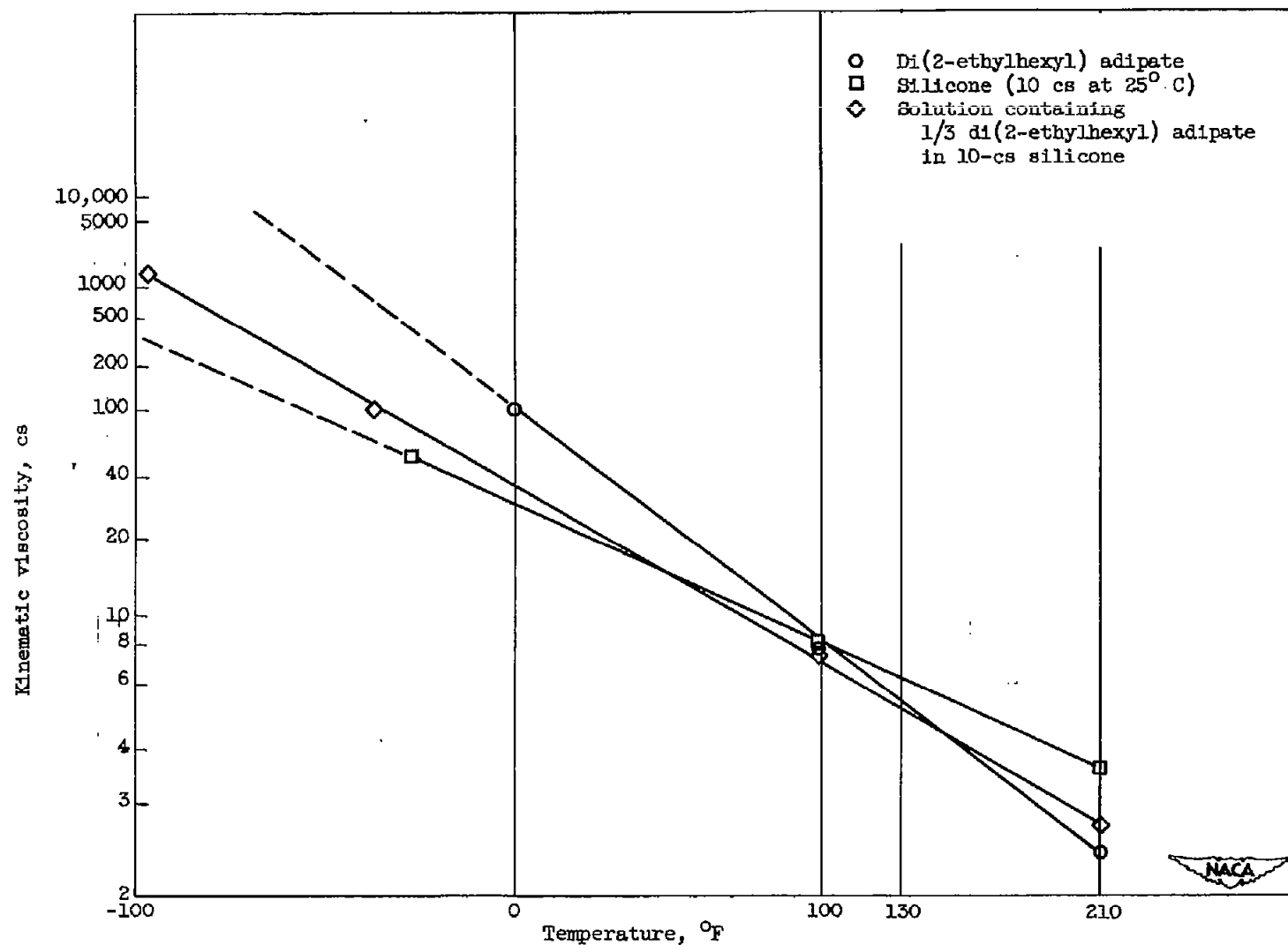loads. Sliding velocity, 120 feet per minute.

Figure 4. - Measured viscosity-temperature relations for
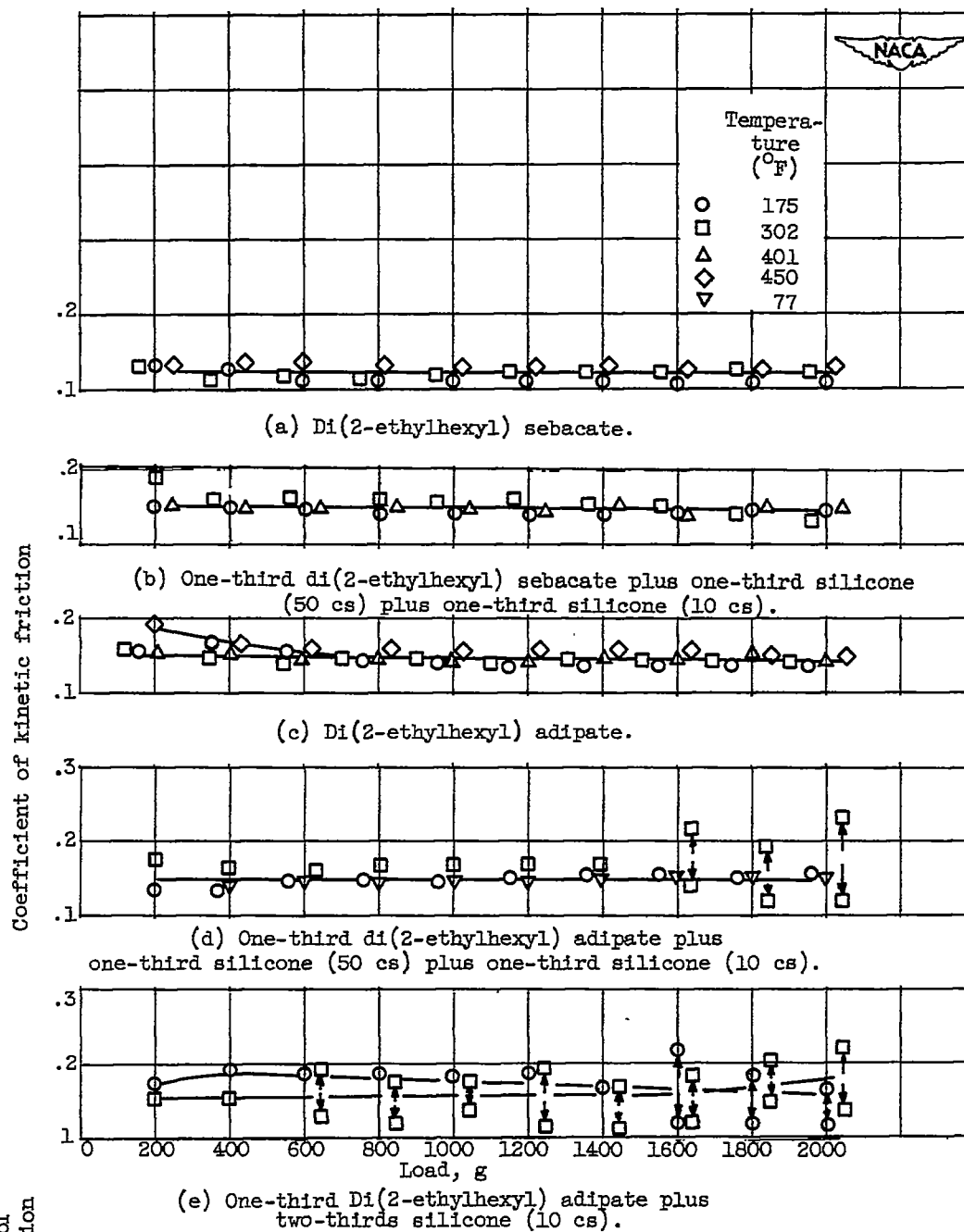diester, silicone, and solution of the two.

(a) Di(2-ethylhexyl) sebacate.

Temperature (°F)

○ 175
□ 302
△ 401
◇ 450
▽ 77

(b) One-third di(2-ethylhexyl) sebacate plus one-third silicone (50 cs) plus one-third silicone (10 cs).

Coefficient of kinetic friction

(c) Di(2-ethylhexyl) adipate.

(d) One-third di(2-ethylhexyl) adipate plus one-third silicone (50 cs) plus one-third silicone (10 cs).

Load, g

(e) One-third Di(2-ethylhexyl) adipate plus two-thirds silicone (10 cs).

Figure 5. - Effect of temperature on coefficient of friction.

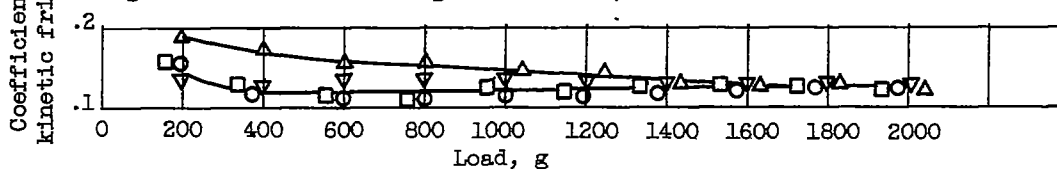Coefficient of kinetic friction

Load, g

Figure 6. - Effect of temperature on coefficient of friction of solution of one-third di(2-ethylhexyl) sebacate and two-thirds silicone fluid (10 cs at 25° C) which contained 2 percent tricresyl phosphate.